THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Thermal Conductivity of Nickel and Other Metals at High Temperatures

R. W. POWELL: 'Thermal Conductivities of Solid Materials at High Temperatures.' *Research*, 1954, vol. 7, Dec., pp. 492-501.

Continuous development of many branches of high-temperature engineering has served to emphasize the necessity for a closer knowledge of the physical properties of metals and alloys at temperatures above normal. In this paper the author has collected, from many sources, thermal-conductivity data for a number of metals and alloys, and has supplemented information available in the literature with results of tests recently made. The introductory section of the review discusses methods used for determination of thermal conductivity, and the main paper is concerned with comparison of data obtained by various workers on commercially pure iron, various types of plain, and low- and high-alloy steel (including many nickel-containing types), non-ferrous alloys (including Monel, 80-20 nickel-chromium alloys and Nimonic 80), polycrystalline oxides, titanium-carbide cermets, carbon and graphite, quartz, silica, rocks, glasses, and other materials.

General trends characteristic of the respective groups of materials are discussed. It is pointed out that the survey reveals certain serious gaps in knowledge, for the filling of which research is in hand, at the National Physical Laboratory and elsewhere.

The review is supported by over 50 *references to the literature*, and is illustrated by numerous curves derived from all available data. A list at the end of the paper classifies the solid materials on which information is available into two groups: electrical conductors and electrical insulators. Attention is directed to the tendency for the thermal conductivities of certain groups of materials to converge towards common values at high temperatures.

Thermal Conductivity of Metals and Alloys at Low Temperatures

R. L. POWELL and W. A. BLANPIED: 'Thermal Conductivity of Metals and Alloys at Low Temperatures. A Review of the Literature.'
U.S. Nat. Bur. Standards, Circular 556, Sept. 1, 1954; 68 pp.*

This publication is issued to satisfy the need for a complete and authoritative compilation of data on thermal conductivity at sub-zero temperatures, the literature of which is widely scattered. Although the treatment is not primarily critical, the authors suggest a method which may be used in evaluation of conflicting data.

The range covered is room temperature to approximately 0°K. (−273°C.), and the information relates to three groups of materials: (1) metals, (2) alloys and steels, and (3) dielectrics. The data include values for materials of varying composition and purity (detailed compositions are given), and in all cases reference to the original source of the information is given. The supporting *bibliography* contains over 200 items.

Nickel-containing materials included in the survey are indicated below:—

- Nickel, pure and commercial grades.
- Corrosion-resisting steels (high-chromium and austenitic nickel-chromium types).
- Low-alloy steels (very little data).
- Nickel-copper alloys (including Monel).
- Nickel-chromium and nickel-chromium-iron alloys.
- Nickel-molybdenum-iron and nickel-molybdenum-iron-chromium alloys.
- Nickel-manganese alloys.
- Nickel-silicon-copper alloys.
- Copper-base alloys containing nickel (including cupronickels, nickel silvers, nickel-containing brasses and bronzes, and nickel-copper-manganese alloys).
- Magnesium-base alloys containing small percentages of nickel.
- Aluminium-base alloys containing small percentages of nickel.

Failure of Metals in Service: Typical Case Histories

J. A. BENNETT and G. W. QUICK: 'Mechanical Failures of Metals in Service.'
U.S. Nat. Bur. Standards, Circular 550, Sept. 27, 1954; 36 pp.*

In the laboratories of the Bureau of Standards, over a long period, many failed service components have been examined and references to individual failures have been made, where appropriate, in publications of the Bureau. In the present Circular thirty-five typical cases are described, as representative of failures most frequently encountered. The examples discussed are static (or overload) fractures, fatigue fractures, and stress-corrosion cracking. Most of the cases quoted relate to parts made from ferrous materials, but failure of some copper- and aluminium-base materials is also included. The report is particularly well illustrated.

The final section embodies some recommendations on precautions which may be taken in design, fabrication, and conditions of use, in order to avoid occurrence of failures of the types described.

* Published by the Superintendent of Documents, U.S. Government Printing Office, Washington, 25, D.C.

NICKEL

Reaction between Nickel and Molten Sodium Hydroxide

R. A. LAD and S. L. SIMON: 'A Study of Corrosion and Mass Transfer of Nickel by Molten Sodium Hydroxide.' *Corrosion*, 1954, vol. 10, Dec., pp. 435-9.

Nickel has been found to be superior to any other material for use as a container for molten sodium hydroxide, but its usefulness is severely hampered at high temperatures by the occurrence of corrosion and mass transfer when a temperature difference is present in the system. The phenomenon of mass transfer is characterized by an almost uniform loss of metal (without intergranular penetration) from the exposed surface in the hot zone, and a deposition of crystalline nickel in the cool zone. A typical example is illustrated.

The authors report some results of an investigation of the nickel/sodium-hydroxide system, which was carried out with a view to elucidating the mechanism of the mass transfer and the factors affecting it. Test equipment and technique are described.

The results show that the amount of mass transfer is strongly affected by variables such as temperature level and temperature gradient, conditions of flow, atmosphere, and the chemical composition of both the sodium hydroxide and the nickel. Relations between rate of mass transfer and temperature gradient were established in the range 1400°–1600°F. (760°–870°C.), and the effects of additions of various substances to the caustic were studied, but on this aspect the results reported are somewhat contradictory.

Determination of Traces of Nickel on Catalysts

G. V. DYROFF and P. SKIBA: 'Determination of Trace Amounts of Iron, Nickel and Vanadium on Catalysts by Fluorescent X-ray Spectrography.' *Analytical Chemistry*, 1954, vol. 26, Nov., pp. 1774-8.

The deleterious effect of minute amounts of metallic elements, especially nickel and vanadium, on cracking catalysts has been recognized for some time in the petroleum industry, but both chemical and spectrochemical methods have failed to give the degree of precision necessary to distinguish between catalysts which, in this respect, could be classed as good or bad. This paper gives full details of a method of X-ray fluorescent analysis which has the following advantages:—it is rapid, requiring only 15 minutes for a complete analysis; the sample required is not large, and can be recovered completely after analysis; a high degree of precision is ensured.

Spectrophotometric Determination of Nickel as the Dimethylglyoxime Complex

O. MENIS, M. L. DRUSCHEL and C. C. SUSANO: 'Spectrophotometric Determination of Nickel as the Dimethylglyoxime Complex.' *Analytical Chemistry*, 1954, vol. 26, Nov., p. 1854.
Abstract of paper to American Chemical Society, October, 1954.

The paper reports modification of the colorimetric method for determination of nickel with dimethylglyoxime, to ensure higher precision and accuracy. Potassium persulphate is used as the oxidizing agent. Optimum conditions for pH, wave-length selection, amount of reagent, tolerance of interfering ions, and time factors are covered in the original paper. Differential spectrophotometric technique was applied to estimation of nickel in steels. The paper also contains a critical review of relevant literature: this reveals conflicting theories with regard to the structure of the nickel-dimethylglyoxime complex.

Determination of Nickel in Presence of Cobalt and Iron

J. H. ROBERTSON and E. E. BYRN: 'Quantitative Separation of Nickel from Iron and Cobalt by Precipitation with Dimethylglyoxime in the Presence of *N*, *N*-Dihydroxyethylglycine as a Masking Agent.' *Analytical Chemistry*, 1954, vol. 26, Nov., p. 1854. Abstract of paper to American Chemical Society, October, 1954.

'Interference of iron (III) and cobalt can be avoided and the determination of nickel made quantitative by precipitation with dimethylglyoxime in the presence of *N*, *N*-dihydroxyethylglycine. This reagent prevents the precipitation of ferric hydroxide even at pH 11 and above. A large excess of the reagent (50 grams or more) prevents complete precipitation of the nickel, but this contingency is simply avoided by the addition of zinc chloride, which forms a complex with the excess of the reagent without releasing the iron or interfering with the nickel precipitation.'

Determination of Magnesium in Nickel

C. L. LUKE and M. E. CAMPBELL: 'Photometric Determination of Magnesium in Electronic Nickel.' *Analytical Chemistry*, 1954, vol. 26, Nov., pp. 1778-80.

The magnesium content of nickel used for the cathodes of vacuum tubes has an important influence on service behaviour and life: accurate control of composition is therefore essential. An analytical method of high sensitivity is required, since magnesium is usually present in only a low range (0·001–0·1 per cent.), and the sample size is frequently strictly limited.

The Titan yellow photometric method has been applied for this purpose, but has proved unsatisfactory, due to inadequate sensitivity and poor reproducibility. In view of the fact that it has been found possible to determine aluminium and several other metals by photometric 8-quinolinol (oxine)—chloroform extraction methods, it appeared likely that such a method could be used for magnesium, after removal of interfering elements. Experiments proved, however, that whereas magnesium can be quantitatively precipitated in ammoniacal solution by 8-quinolinol, it is very incompletely extracted from ammoniacal solution by a solution of 8-quinolinol in chloroform. Further experiments were therefore carried out to find a suitable water-immiscible solvent for the magnesiumquinolate. Only acetophenone was found to be a good solvent for the magnesium

compound, and this had to be rejected due to non-reproducibility of results and the objectionable odour resulting from its use.

The possibility of improving the chloroform extraction was next considered, and it was found that this can be done by addition of a water-miscible organic solvent to the aqueous solution. In the method now proposed, of which full working details are given, interfering elements are removed by hydroxide, oxalate, sulphide and carbamate separations, butyl Cellosolve and ammonium hydroxide are added, and the magnesium is extracted into a solution of 8-quinolinol in chloroform, and is photometrically estimated.

Charging of Nickel-Alkaline Batteries

'Automatic Charging of Nickel-Iron Batteries.' *Engineering*, 1955, vol. 179, Jan. 8, p. 27.

This brief note refers to an automatic charging system which has been developed for use on the 60-cell nickel-iron batteries with which the 34 electric vans operated by Messrs. Harrods, Ltd. are equipped.

Nickel Chelates

H. C. CLARK and A. L. ODELL: 'The Properties of Some Nickel Complexes in Solution.' *Chemistry and Industry*, 1954, Dec. 4, pp. 1510-1.

An extensive investigation is being made, in the Department of Chemistry, Auckland University College, on nickel chelates formed with salicylaldehyde derivatives. This note gives some preliminary results; full details are to be published later.

Thermal Conductivity of Metals and Alloys at Low Temperatures

See abstract on p. 26.

ELECTRODEPOSITION AND OTHER COATING METHODS

Electrodeposition of Nickel from Sulphamate Baths

M. B. DIGGIN: 'Nickel Plating from Sulphamate Baths.' *Metal Progress*, 1954, vol. 66, Oct., pp. 132-7.

The subject matter of this paper is closely similar to that of a fuller paper presented at the Fourth International Conference on Electrodeposition and Metal Finishing in April, 1954; see abstract in *The Nickel Bulletin*, 1954, vol. 27, No. 5, p. 85.

Electrodeposition of Alloys

A. WHITTAKER: 'Electrodeposition of Alloys.' *Machinery (Lond.)*, 1954, vol. 85, Dec. 24, pp. 1350-3.

This article is of a review character. The author first gives a brief outline of the advantages and scope of alloy plating, the types of alloys which can be electrodeposited, the essential principles of alloy deposition, and the general influence of process variables. These notes are followed by a succinct summary of the main features of alloy-deposition processes which are now being worked: tin-nickel, indium-zinc, indium-copper, lead-silver, nickel-cobalt, and lead-tin. Typical solutions used, conditions for electrodeposition, and the uses of the respective types of alloy plating are given.

Electroless Plating: Review of Development

A. BRENNER: 'Electroless Plating Comes of Age.' *Metal Finishing*, 1954, vol. 52, Nov., pp. 68-76; Dec., pp. 61-8.

This review comprises a survey of development from 1946, when the present author (the originator of the method) announced the results of his work at the U.S. National Bureau of Standards, to the present day, when the process is in commercial development in several countries. The review is based on (1) the literature, of which details are given in a *bibliography* of some 50 references, and (2) commercial practice and experience. In order to provide a general background, the paper is introduced by discussion of the main features of the process, for which the basic patents are U.S. 2,532,283 (deposition of nickel) and 2,532,284 (deposition of cobalt). These are public patents, and royalty-free licences are obtainable from the Department of Commerce, Washington, D.C.

The scope of the survey is indicated below:—

The Electroless Process. To date only nickel, cobalt or nickel-cobalt alloys have been deposited in a manner capable of practical application. The nature of the baths used, the chemical reactions involved, the variables affecting rate of deposition, and the properties of the deposits are outlined. Reference is directed to operating instructions which are available from the Bureau of Standards. The discussion of properties includes a table comparing the hardness, at various temperatures, of electroless and of electrodeposited nickel coatings.

The Literature. It is pointed out that in spite of the numerous articles on electroless plating which have appeared since 1947, there is no research paper in a technical journal. The most informative items, from the point of view of progress, are some half dozen patents, the main points of which are discussed in some detail in this review.

Present Status of the Process. A survey of industrial utilization of electroless plating disclosed that it is being used on a small scale for deposition of nickel, and in several applications is meeting a felt need. It is not known whether commercial deposition of cobalt or nickel-cobalt alloys is being carried out, although licences have been issued for such purposes. In the user group the most important firm is General American Transportation Company. This Company has carried out much research, and, in addition to patents already granted, has nearly twenty pending applications relating to improvements in the process, which is known as 'Kanigen' (catalytic nickel generation). Attention is also directed to Metal Processing Company, Inc., who have used the designation 'Lustraloy' to describe their deposits. Information

on current methods of operation is based on experience of the Eastman Kodak Company, North American Aviation, Inc., General Motors Corporation, Curtiss-Wright Corporation, General American Transportation and others.

The review of current practice is made in sections dealing with the following aspects:—

Size of Installation and Type of Work being handled

Plants of various sizes are described. Typical parts being treated include vacuum-pump equipment, gears, tubes, moulds, aircraft components, tank interiors, reaction vessels, wall-tiles, and many types of recessed parts.

Equipment for Electroless Plating

Notes on materials of construction for tanks and auxiliary equipment.

Pre-treatment before Plating

Cleaning treatments normal in plating shops are used by most operators.

Bath: Composition, Purification, Life, and Rate of Plating

Most of the plants use the acid baths; two small installations employ the alkaline type. Detailed notes are given on addition media. Commercial-grade salts are used in most cases; details are given of purification methods, for use where required. The only satisfactory method for evaluation of the life of the bath on an economic basis is with respect to the amount of nickel which has been deposited from it: reports on typical experience of life are summarized. Rates of deposition from (*a*) acid, and (*b*) alkaline baths are discussed: plants operating on a production basis give rates varying from 0·2 to 0·8 mil per hour, the greater proportion being towards the lower end of this scale. The gradually decreasing rate of deposition with time is a limitation in the electroless process, and appears to be a subject for research. The occurrence of spontaneous decomposition of the bath, and means for prevention of this deterioration, are considered.

Operating Conditions

pH of 4–6 is generally favoured. Rate of deposition is highly sensitive to temperature of the bath: 195°–200°F. (90°–93°C.) is the most usual range, but probably there is no closely critical temperature. Agitation of the solution is considered necessary by some operators. Replenishing with nickel salts and hypophosphite is generally practised: particular reference is made to the method used by the General Electric Company.

Deposition on Non-Catalytic Surfaces

Methods which have been evolved, by the authors and others, are briefly summarized, with particular reference to techniques for electroless plating of glass and plastics.

Electroless Deposition of other Metals

Attempts to develop electroless chromium plating have been unsuccessful; silver deposits so produced comprise a mossy precipitate, not a metallic plate; exploratory deposits in the author's laboratory have been partially successful with rhodium. Preliminary work on deposition of other metals is also noted.

Economics of the Electroless Plating Process

The key to the economics of the process is stated to be the cost of the hypophosphite: this and other variables are considered, and it is concluded that, while electroless plating is not generally competitive to electrodeposition, there are certain specialized applications for which it is uniquely suitable and likely to be commercially employed.

Determination of the Hypophosphite Content

The importance of control of this variable is emphasized, and particulars are given of methods employed by various users of the electroless process.

Electroless Nickel: Alcoplate

'Nickel Plating without Electrodes.'
Chemical and Engineering News, 1954, vol. 32, Dec. 13, p. 4922: see also *American Metal Market*, Dec. 1, 1954.

Alcoplate is the name given by American Locomotive Company to their electroless plating process, which has been adapted from the Kanigen process (see previous abstract). This short note describes some of the features of the large continuous-plating plant which has been erected by the American Locomotive Company. Among other applications, Alcoplate is being used for coating the surfaces of reaction vessels used by the Atomic Energy Commission. It is claimed that the corrosion-resistance of the coating (93 per cent. nickel, 7 per cent. phosphorus) is superior to that of nickel.

Electroless Nickel Plating: Service Experience

J. D. MACLEAN and S. M. KARTEN: 'A Practical Application of Electroless Nickel Plating.'
Plating, 1954, vol. 41, Nov., pp. 1284–7; disc., p. 1287.

Full reprint, with discussion, of paper to which brief reference was made, from an abstract source, in *The Nickel Bulletin*, 1954, vol. 27, Nos. 7–8, p. 129.

The paper deals with the practical application of the process to steel components of bomb fuses: it was selected for treatment of these parts because of the ability of the type of bath used to plate evenly to the bottom of blind holes. A diagram of the fuse rotor embodying these holes is shown in the report, and other parts for which basket plating by the electroless method has been successfully used are also illustrated.

Several solutions were described by the originators of the process (BRENNER and RIDDELL, of the U.S. National Bureau of Standards): for the purpose with which the present authors were concerned the citrate type of bath was chosen on account of the brightness of the plating obtainable from it, and the ease with which it can be controlled. Rate of deposition, which is slower with this solution than with some of the alternative baths, was speeded up by increasing the concentrations of nickel and hypophosphite. Maintenance of composition was ensured by the use of a replenishing stock solution of high nickel content.

To prevent build-up of small nickel particles precipitating out of solution, the tanks are decanted daily and treated, under a hood, with 50 per cent. nitric acid. 2½-gallon white stoneware tanks were found to be ideal. The plating lines are set up as complete units, each providing for a hot electric cleaner, cold electric cleaner, acid dip, two nickel-plating tanks, two rinse tanks with overflow dams, a centrifugal drier, and a degreaser. The baskets are of stainless-steel mesh, with stainless-steel rivets and handles. They build up gradually with nickel, but are stripped once a week in the 50 per cent. nitric acid solution. Nickel plating is carried out at 195°F. (90°C.). The stoneware tanks are set in a water bath consisting of a stainless-steel tank, in which are two immersion heaters. Ventilation is accomplished by means of stainless-steel hoods which curve round one side of the plating tank and are connected to an exhaust fan. (The set-up is illustrated). The following details are given of routine procedure:—

(1) Hot alkaline cleaning in a 4 oz./gal. solution; reverse current; 4 volts; 15 amp; 180°F. (85°C.); 4 minutes' treatment.

(2) Rinsing in running water.

(3) Cleaning in cold caustic cyanide solution: KOH 4 oz./gal.; NaCN 4 oz./gal.; D.C.; stainless-steel anodes; 4 volts; 15 amp; room temperature; 4 minutes' treatment.

(4) Rinsing in running water.

(5) Acid dipping; 20 per cent. H_2SO_4; room temperature; treatment for 10–20 seconds.

(6) Rinsing in running water.

(7) Nickel plating; 195°F. (90°C.), for 8 minutes.

(8) Rinsing in running water.

(9) Final rinsing; water + 2 oz./gal. of a wetting agent.

(10) Drying for 3 minutes in a centrifugal drier.

or

as an alternative drying method, two methanol dips may replace operations (9) and (10).

NOTE.—The baskets are agitated in the cleaning solutions and in the nickel-plating bath by shaking for two or three seconds once a minute.

Solutions Required

	oz./gal.
A. *New Nickel Solution*	
Nickel chloride, $NiCl_2.6H_2O$	6·0
Ammonium chloride, NH_4Cl	6·7
Sodium citrate, $Na_3C_6H_5O_7.5H_2O$ ..	13·4
B. *Replenishing Solution*	
Nickel chloride $NiCl_2.6H_2O$	20·0
Ammonium chloride, NH_4Cl	2·0
Sodium citrate, $Na_3C_6H_5O_7.5H_2O$ ..	6·0
C. *Hypophosphite Solution*	
Sodium hypophosphite, $NaH_2PO_2.5H_2O$	60

In all cases the chemicals listed are dissolved in water, and the solutions are filtered.

D. *Ammonia Solution*

One part of concentrated ammonia water is diluted with two parts of water. The new nickel solution is heated to 195°F. (90°C.) and 3 fluid ounces of the hypophosphite solution are added for every gallon of nickel solution used. Ammonia solution is added until the first permanent blue colour is obtained: this will represent a pH of 8·5–9·0, and the solution is then ready for use.

Since the nickel plates out of the solution, replenishment is essential. Approximately 4 fluid ounces of replenishing nickel solution per gallon of bath should be added during each hour of operation. Restoration of the composition to the correct nickel concentration is indicated by the appearance of a white precipitate on further addition of replenishing mixture. Consumption of the hypophosphite is compensated by addition, every half hour, of approximately 1 fluid ounce of hypophosphite solution per gallon of nickel solution.

Moderate gassing of the work being plated should occur at all times; slowing down of gassing is an indication of low hypophosphite content; excess hypophosphite will result in too violent gassing, which may precipitate all the nickel in the solution. When sufficient nickel and hypophosphite is present, ammonia is added, during maintenance, as at the start, i.e., till the first permanent blue is observed.

It is pointed out that for the type of work for which it is suitable, electroless nickel plating has the advantage of uniformity, brightness, and smoothness, with freedom from pitting and burning. Up to a thickness of 0·00007 in. the authors have observed no sign of brittleness in the coatings. Many parts which would require racking for electroplating can be successfully treated in a basket by the immersion process, effecting a saving of time and labour which is significant in quantity production.

NON-FERROUS ALLOYS

Solid Solubility of Nickel in Germanium: Diffusion Studies

F. VAN DER MAESEN and J. A. BRENKMAN: 'The Solid Solubility and the Diffusion of Nickel in Germanium.' *Philips Research Reports*, 1954, vol. 9, July, pp. 225-30.
Abstract in *Wireless Engineer*, 1954, vol. 31, Dec., Abs. 3589.

'Nickel produces rapidly diffusing acceptors in germanium. Measurements of the Hall effect and of resistivity show that the nickel acceptor level lies 0·23 eV above the valence band. On this basis solid solubility of nickel in germanium, between 700° and 900°C., is derived from resistivity determinations, and the distribution and diffusion coefficients are calculated. Diffusion coefficient of nickel in germanium was measured between 700° and 850°C. The distribution coefficient is $1{\cdot}8 \times 10^{-6}$ at the melting point of

germanium. The activation energy of diffusion is 21 kcal/mole. Annealing of a nickel-saturated germanium sample restores the original resistivity.'

Porosity in Nickel-Bronze Casting

'Leaky Nickel-Bronze Castings.'
Metal Industry, 1954, vol. 85, Dec. 10, p. 485.

Report of examination of flanged nickel-bronze casting reported to leak under pressure test. The casting had the following composition: copper 63·15, nickel 22·1, tin 5·64, zinc 4·94, silicon 0·52, lead 0·83, iron 1·52, aluminium 0·16, manganese 1·01, phosphorus trace, sulphur 0·06, per cent. The porosity is considered to have been due to unfed shrinkage, and full recommendations are made for procedure which should eliminate the defect.

Nickel and Nickel-Alloy Welding Electrodes: A.S.T.M. Specification

AMER. SOC. TESTING MATERIALS: 'Tentative Specifications for Nickel and Nickel-base Alloy Covered Welding Electrodes.'
A.S.T.M. B 295-54*T*: *A.W.S. A*5.11-54*T*.
1954 *Supplement to Book of A.S.T.M. Standards, Part* 2, *Non-Ferrous Metals*, pp. 277-88.

The specifications cover nickel and nickel-base alloy electrodes for shielded metal-arc welding of nickel and nickel-base alloys individually to themselves, for welding these materials to steel, and for welding the clad side of nickel-base-alloy-clad steels.

The requirements cover chemical composition of ten types of weld metal deposited by the procedures specified, and tests to be applied to the welded joints. An appendix contains a guide to the A.W.S./A.S.T.M. classifications of the types of electrode covered by the specification, describes the nature and intended use of the respective electrodes, and gives details of some special tests for electrodes (corrosion, scaling, mechanical and radiographic).

Thermal Conductivity of Nickel and Other Metals at High Temperatures

See abstract on p. 26.

Thermal Conductivity of Metals and Alloys at Low Temperatures

See abstract on p. 26.

Corrosion-Resistance of Cupro-Nickel Alloys

See abstract on p. 38.

Materials for Handling Sulphuric Acid and Acid Sludges in Refineries

See abstract on p. 37.

Nickel-containing Materials in Shafts

See abstract in next column.

Failure of Metals in Service: Typical Case Histories

See abstract on p. 26.

NICKEL-IRON ALLOYS

Flux Resetting Characteristics of Magnetic Nickel Alloys

H. HUHTA: 'Flux Resetting of Several Magnetic Materials.' *Electrical Engineering*, 1954, vol. 73, Oct., p. 908.

(Abstract of paper 54-161, presented before the American Institute of Electrical Engineers, January, 1954.)

The paper deals with the flux resetting characteristics of some magnetic materials commonly used in amplifiers:—Deltamax, 65-Permalloy, Supermalloy, and Perminvar. The alloys were used in various physical conditions.

In the experiments described, the flux was forced to change under the influence of a constant external magnetizing force. The operation of a core employed in self-saturating magnetic amplifier circuits with high-impedance control windings approximates these conditions during the non-conducting half-cycle of supply voltage: the data obtained in this investigation are therefore readily applicable to the study of that type of magnetic amplifier.

All the tests reported were on gapless tape-wound toroidal cores made of commercially available materials. A special test circuit was constructed to drive the flux from saturation in one direction to saturation in the opposite direction with a constant current through one winding on the core. The induced voltage in another winding was displayed on an oscilloscope, to give the rate of change of flux as a function of time, and the oscilloscope display was photographed, to provide a record of the waveshape, and to permit determination of peak and average rate of change of flux. Typical results are illustrated.

The observations made indicate that it is reasonable to expect the highest gain from a self-saturating magnetic amplifier by choosing core materials which give the highest value of the ratio of (rate of change of flux)/(saturation flux density times excess magnetizing force)*. Although, for various reasons, this criterion is not precise, it is considered feasible to use it to assist in evaluation of core materials. The results obtained varied too widely to permit of their safe use for design purposes.

CONSTRUCTIONAL STEELS

Nickel-containing Materials in Shafts

C. R. WEBB: 'Shafts in Alloy Steels.'
Mond Nickel Co. Ltd. Publn. 367*A*, 1954; 26 pp. (Reprint of Publn. 367, with some revision.)

The information given is primarily intended to assist engineers in designing shafts in direct-hardening and case-hardening nickel-alloy steels. In view of the fact that corrosion-resistance is also in some cases

* The term excess magnetizing force is defined as the test magnetomotive force (as calculated from the current and turns) minus the D. C. coercive force.

an important property in shafts, data on Monel, corrosion-resisting steels, and Inconel have been included.

The author discusses the nature of the stresses which occur in shafts, strength and other considerations governing choice of materials, and design of shafts for operation under various conditions of stressing.

In reviewing materials, the compositions, treatments and mechanical properties of suitable nickel-alloy steels are shown in tables and figures, and working stresses and other relevant data are tabulated for Monel, corrosion-resisting steels and Inconel.

Finally, some notes are added on parts and devices commonly used in association with shafts, e.g., bearings, couplings, keys, splines, etc.

Determination of Molybdenum in Steels

BRIT. IRON AND STEEL RESEARCH ASSOCN., METHODS OF ANALYSIS COMMITTEE: 'Absorptiometric Determination of Molybdenum in Iron and Steel.'
Jnl. Iron and Steel Inst., 1954, vol. 178, Dec., pp. 356–9.

Existing gravimetric and colorimetric methods for determination of molybdenum in tungsten-bearing steels have not proved entirely satisfactory, due to co-precipitation of molybdenum with tungsten. The method recommended in B.S. 1121: Part 26, published in 1952, covers only steels containing up to 0·5 per cent. of tungsten.

The investigation outlined in the present report was made with the object of providing improved absorptiometric procedure (based on formation of a molybdenum thiocyanate compound) suitable for analysis of a wide variety of molybdenum-containing steels. In the method recommended, formation of coloured reaction products which interfere, including those of iron, tungsten, cobalt, vanadium, and copper, is suppressed or is compensated by means of correction graphs. Titanium, added to the final solution, catalyzes the reaction, and ensures complete reduction of the ferric thiocyanate. Full details of procedure are given. The importance of obtaining complete decomposition of samples is emphasized.

The method has been successfully applied to a variety of iron-base materials, including austenitic steels of the 18–8 chromium–nickel type. Typical results on the various classes of steel, obtained by eleven different analysts, are quoted in the report.

Thermal Conductivity of Nickel and Other Metals at High Temperatures

See abstract on p. 26.

Thermal Conductivity of Metals and Alloys at Low Temperatures

See abstract on p. 26.

Failure of Metals in Service: Typical Case Histories

See abstract on p. 26.

HEAT- AND CORROSION-RESISTING MATERIALS

Effect of Cyclic Heating and Stressing on the Properties of Metallic Materials

AMER. SOC. TESTING MATERIALS: 'Symposium on Effect of Cyclic Heating and Stressing on Metals at Elevated Temperatures.'
A.S.T.M. Special Technical Publn. 165, 1954: 175 pp.

This report contains reprints of papers and discussions presented at the 57th Annual Meeting of the Society, in June, 1954. The symposium was sponsored by the A.S.T.M./A.S.M.E. Joint Committee on Effect of Temperature on the Properties of Metals.

The majority of the papers have been abstracted in *The Nickel Bulletin*, from preprints: references to such abstracts are given below, in brackets after the titles of the individual contributions. Papers to which no earlier reference has been made are briefly reviewed in this issue.

D. FREY: 'Introduction,' p. 1.
Brief notes on the aims of the symposium and its scope.

J. E. DORN and L. A. SHEPARD: 'What We Need to Know about Creep', pp. 3–28; disc., pp. 28–30.

It is urged that a new outlook is required on the problems of creep, and that progress must be hoped for by a more intimate investigation of the basic laws governing it. An attempt is made to refute objections which may be raised to such views as being unpractical. The authors propose a method of approach providing for isolation and separate study of the effects of the individual variables of temperature, stress, and structure of the metal. Results obtained by the methods proposed, on some pure metals and some dilute alloys, are recorded; problems requiring further clarification are discussed, and methods are suggested by which the study of creep in complex alloys, and creep at relatively low temperatures, may be advanced.

L. F. COFFIN: 'The Problem of Thermal Stress Fatigue in Austenitic Steels at Elevated Temperatures', pp. 31–50; disc., pp. 50–1.
(*Nickel Bulletin*, 1954, vol. 27, Nos. 7–8, p. 142.)

J. MILLER: 'Effect of Temperature Cycling on the Rupture Strength of Some High-Temperature Alloys', pp. 53–65; disc., pp 65–6.
(*Nickel Bulletin*, 1954, vol. 27, Nos. 7–8, p. 142.)

G. V. SMITH and E. G. HOUSTON: 'Experiments on the Effects of Temperature and Load Changes on Creep Rupture of Steels', pp. 67–76; disc., pp. 77–8.
(*Nickel Bulletin*, 1954, vol. 27, No. 10, p. 197.)

R. H. CAUGHEY and W. B. HOYT: 'Effects of Cyclic Overloads on the Creep Rates and Rupture Life of Inconel at 1700° and 1800°F',
pp. 79–102; disc., pp. 103–4.
(*Nickel Bulletin*, 1954, vol. 27, No. 10, p. 196.)

G. J. GUARNIERI: 'The Creep-Rupture Properties of Aircraft Sheet Alloys Subjected to Intermittent Load and Temperature', pp. 105-46; disc. pp. 147-8.

The author points out that it would be impractical, if not impossible, to conduct intermittent load and heat tests equivalent to all the variations and sequence of stress and temperature typical of aircraft operation. The alternative is to establish general trends, utilizing a simplified series of cyclic test conditions so selected as to permit useful interpretation of the data. Such a principle was adopted in the tests described in this paper.

High-temperature creep-rupture properties of six sheet alloys of interest to the aircraft industry were investigated (*a*) under the cyclic conditions given below, and (*b*) under constant-temperature, constant-load conditions. The materials examined are listed in the following Table:—

Materials	*Test Temperatures*
Titanium–stabilized 18–8 chromium–nickel steel	1200° and 1350°F. (648° and 735°C.)
N–155 Multimet alloy	1350° and 1500°F. (735° and 815°C.)
Inconel 'X' (precipitation–hardenable nickel–chromium–iron–base alloy)	1350° and 1500°F. (735° and 815°C.)
RC–130–A titanium	800°F. (425°C.)
24S–T3 Alclad aluminium	300°, 450° and 600°F. (150°, 232° and 315°C.)
Magnesium (containing 2·5–3·5% aluminium)	300° and 450°F. (150° and 232°C.)

Intermittent-load/constant-temperature tests were made, in most cases, with a cycle of one hour load-on, one hour load-off: several of the alloys were also subjected to a cycle involving eight hours load-on, eight hours load-off.

Intermittent-temperature/constant-load tests comprised a two-hour cycle, with the specimen at temperature for one hour and cooled for one hour.

The data recorded show that a wide variety of effects was produced by the intermittent-load and -temperature conditions. Stability of microstructure was one of the most significant variables. In cases in which overageing, relaxation, recrystallization and loss of ductility occurred, acceleration of creep and rupture was induced by intermittent loading. Retardation of creep and rupture was produced in alloys in which increase in ductility and creep recovery resulted from the intermittent-load cycle. Intermittent heating produced acceleration of creep and rupture in a large number of the tests, particularly where susceptibility to intergranular oxidation and cracking was aggravated by thermal stresses.

W. F. SIMMONS and H. C. CROSS: 'Constant and Cyclic-Load Creep Tests of Several Materials', pp. 149-72; disc. p. 172.
(*Nickel Bulletin*, 1954, vol. 27, Nos. 7-8, p. 143.)

D. N. FREY: 'Summary of Outstanding Points in the Papers Presented', pp. 173-5.

Formation of Carbide, Sigma and Chi Phases in Molybdenum-containing Austenitic Steels

H. C. VACHER and C. J. BECHTOLDT: 'Delta Ferrite-Austenite Reactions and the Formation of Carbide, Sigma and Chi Phases in 18 Chromium-8 Nickel-3·5 Molybdenum Steels.'
Jnl. of Research, Nat. Bur. Standards, 1954, vol. 53, Aug., pp. 67-76; R.P. 2517.

The compositions of the three steels in which the phenomena were studied are shown below:—

Table I
Compositions of Steels Studied

C %	Si %	Mn %	Ni %	Cr %	Mo %
0·05	0·48	0·71	7·9	17·6	3·2
0·08	0·49	1·65	9·4	17·8	3·6
0·07	0·68	1·58	13·5	18·4	3·6

Solution treatments were varied, in order to obtain δ and γ phases of different compositions, and δ in varying amounts. This procedure takes advantage of the fact that for a certain steel the chemical composition of the reacting phases at equilibrium varies with temperature, and that equilibrium concentrations are approximated in the solution treatment. It is not possible to vary the relative amounts of δ and γ and maintain their composition constant by heat-treatment because, like composition, the relative amounts vary with temperature. The δ-γ reaction was studied both in the presence and in the absence of the γ_S phase*. This was done by separating the δ phase from the solution-treated steels and reheating it in the range 1200°-1700°F. (650°- 925°C.), along with the solution-treated steels. Table II on p. 34 shows the re-heating treatments.

The constitution of the re-heated ferrites and of the solution-treated steels was determined by metallographic study. Full details of experimental technique and many typical photomicrographs and X-ray diffractometer charts (obtained from residues) are given in the paper.

The observations made lead to the following conclusions:—

Examination of the heat-treated specimens showed four fields of stability: a region in which γ was the only phase; a region in which γ and δ were stable; a region in which γ, carbide ($M_{23}C_6$) and σ were stable, and a region in which γ, carbide ($M_{23}C_6$), σ, and χ were stable.

The δ was separated from the γ and heated at the lower temperatures, along with samples of the parent steels. The chemical analyses of δ and the computed analyses of γ indicated that the distribution ratios for chromium, nickel, molybdenum and manganese in the δ/γ phases were approximately 1·2, 0·5, 1·7, and 0·9 respectively, in the range 1800°-2500°F. (980°-1370°C.).

Results showed that the decomposition of δ and

*γ_S=γ formed during solution treatment, as distinct from that formed at lower re-heating temperatures, which the authors designate γ_R.

Table II*

Solution Treatment				Re-heat Treatment			
Identification	Temperature		Period	Identification	Temperature		Period
	°F.	°C.	hrs.		°F.	°C.	hrs.
A	2500	1370	½	K	1700	925	340
B	2000	1095	½	L	1500	815	500
C	1800	980	18	M	1200	650	500
†D	2250	1230	18				
†E	1900	1035	2				

* All specimens were water-quenched from all temperatures. † Steels 2 and 3 only: see table on p. 33

the kind of products that were formed were influenced by small differences in the chromium, nickel and molybdenum contents.

Examination of the re-heated steels showed that the amount of δ formed in the solution treatment affected the decomposition of δ and the kind of products formed at certain re-heating temperatures. It appeared that there was a tendency for the reactions to nucleate in the δ sites and to continue by diffusion of elements from the matrix. When the volume of decomposition products exceeded the volume of the initial δ, the products tended to be distributed intragranularly.

Thermal Conductivity of Nickel and Other Metals at High Temperatures

See abstract on p. 26.

Thermal Conductivity of Metals and Alloys at Low Temperatures

See abstract on p. 26.

Nickel-containing Alloys for Brazing of Titanium and Titanium Alloys

R. A. LONG and R. R. RUPPENDER: 'High-Temperature Alloy Fusion Brazing for Titanium and Titanium Alloys.' *Welding Jnl.*, 1954, vol. 33, Nov., pp. 1087–90.

Brazing of titanium and high-titanium alloys has been carried out until now by using pure silver as the brazing medium, a suitable flux, and induction heating. The use of silver-base materials would, however, be almost impossible in furnace brazing, since the time cycle essential for obviating formation, by diffusion, of brittle layers of intermediate phases would be too short to be commercially practicable. It was therefore recognized that a new series of brazing alloys must be developed which would (*a*) have a flow temperature within the metallurgical limitations of the titanium-base metal, and (*b*) not alloy by a strictly diffusion process.

Ti %	Ni %	Cu %	Co %
28	52	15	5
64·35	25·65	10	0

The work described in this paper has revealed that some fifteen titanium-nickel alloys are promising for this purpose. Two of these (see table in the left-hand column) have been partially evaluated.

Results to date indicate that alloys utilizing primarily the eutectics of the titanium-nickel system are suitable for use in the 'fusion' type of joining, operating under gas-shielded furnace-brazing conditions.

Argon was used by the authors for the experiments reported. Compressive and tensile shear strengths of more than 40,000 p.s.i. (18 tons per sq. in.) have been obtained. By means of such alloys, flow temperatures of 1700°–1900°F. (925°–1035°C.) have proved to be possible, and temperatures in this range can be used without damaging commercially pure titanium.

Rare-Earth Additions to Stainless Steels

H. O. BEAVER: 'Rare Earths in Stainless Steels.' *Metal Progress*, 1954, vol. 66, Oct., pp. 115–19.

This paper is based on experience in the works of Carpenter Steel Company, Reading, Pa., who are using rare-earth additions for improvement of hot-workability of stainless steels of types which were not previously considered hot-workable in a commercial sense. Specific reference is made to the success of this technique in a high-nickel-chromium steel of the Carpenter 20 type, in which the copper present, although enhancing resistance to acid corrosion, vitiates hot-workability.

Attention is directed to detailed discussions of this subject which have been presented by the author and others in various technical papers, and a survey is made of results achieved to date and of conclusions drawn from laboratory experiments and large-scale production. 'Rare-earth metals' are defined, and the results obtainable by additions of these metals and their oxides are discussed. Experience with this technique has shown that:—

(1) Mischmetall additions made to high-alloy stainless steels which are inherently hot-short can transform them into hot-ductile materials. The

cerium + lanthanum content recommended is 0·02 per cent. minimum.

(2) Smaller additions of Mischmetall, to lower-alloy stainless steels, e.g., of the 18-8, 25-20, and 18-11-Mo types, which are inherently ductile, improve hot-workability as gauged by higher yield of prepared billets or slabs.

(3) Rare-earth oxide mixtures may improve the hot-workability of inherently ductile austenitic stainless steels (as evidenced by higher yields of prepared billet) and the cost of such treatment is significantly less than that involved in the use of Mischmetall. Rare-earth oxide additions do not, however, improve the hot workability of essentially hot-short types of austenitic stainless steel, or those which are difficult to hot-work, although residual cerium and lanthanum in amounts of 0·02-0·025 per cent. is usually beneficial.

The exact mechanism of the effect of the rare earths and rare-earth oxides is not yet clear, but it has been established that such treatment lowers the oxygen content of the steels, and that rare earths have a definite effect on sulphur content. It has also been suggested that rare-earth additions tend to refine the grain-size of the steels, but the present author and his colleagues record their conviction that although this may be a factor in increasing yield in inherently ductile steels, mere refinement of grain size is not of significance in converting inherently hot-short austenitic stainless steels into commercially hot-workable materials. It is considered possible that there may also be some effect of the rare-earth additions on neutralization of the effects of such tramp elements as might deleteriously influence hot-workability, but the evidence on this point is not yet conclusive. A high hydrogen content can nullify the effect of Mischmetall, but simple removal of hydrogen does not in itself produce the desired ductility. Experience has shown that a definite residual rare-earth content is essential mainly in the complex austenitic nickel-chromium steels which contain molybdenum and/or copper.

After this summary of the effects of rare earths and their oxides on hot-workability, the author reviews the influence of such additions on other properties.

(1) *Properties not affected*

Tests made under standard conditions have shown that *corrosion-resistance* is unaffected (as gauged by the Huey nitric-acid test); *tensile strength* of annealed austenitic steels is unchanged (both at normal and elevated temperatures); *ductility* at room temperature is only slightly, but favourably, affected; *impact* is substantially unaffected, although with Mischmetall additions some slight improvement may result. In *age-hardening* experiments neither beneficial nor detrimental effects have been found to result from rare-earth or rare-earth-oxide additions.

(2) *Properties affected*

Stress-rupture values are definitely raised by treatment with rare earths: typical values are given. *Elongation and reduction of area* for stainless steel containing Mischmetall have also been observed to be higher at given stress and temperature than for similar steels treated with rare-earth oxides or untreated. The influence of the additions on *resistance to scaling* is believed to be largely dependent on the composition of the basis steel: a beneficial effect of Mischmetall has been found in scaling tests up to 1900°F. (1035°C.), but above this temperature scaling increased rapidly. Such reversal is at present unexplained.

High-Chromium, Low-Nickel 'Intermediate' Stainless Steel

A. J. LENA: 'New Stainless Alloy Bridges Gap between 300 and 400 Series.' *Iron Age*, 1954, vol. 174, Dec. 2, pp. 113-16.

This article describes a steel developed, by Allegheny Ludlum Corporation, to give properties intermediate between those of the straight-chromium (A.I.S.I. 400 series) and the fully austenitic nickel-chromium steels (A.I.S.I. 300 series). The composition of the new steel, designated A.M. 350, is as follows: carbon 0·08, silicon 0·40, manganese 0·60, chromium 17·0, nickel 4·2, molybdenum 2·75, per cent. The optimum properties of the steel are developed by refrigeration, followed by tempering: alternatively, it may be hardened by a double ageing, but with some loss in mechanical properties and corrosion-resistance. In the annealed condition (annealing temperature 1700°-1900°F.: 925°-1035°C.) the high-chromium, low-nickel steel has properties similar to those of conventional austenitic steels, but on cooling to sub-zero temperatures (−80° to −100°F.: −62° to −73°C.) transformation to martensite occurs. Maximum strength and ductility, combined with a useful degree of corrosion-resistance, are achieved by tempering at 750°F. (400°C.) for about two hours: such treatment has the added advantage that no problem of scaling is involved. The alternative (double-ageing) treatment which may be used comprises heating for one hour at 1300°-1350°F. (705°-735°C.), followed by one hour at 850°F. (455°C.).

Room-temperature and short-time high-temperature properties typical of the steel in both conditions are given, and comparison is made with corresponding properties characteristic of straight-chromium steels, showing the usefulness of the nickel-containing type.

A.M. 350 has a high capacity for work-hardening: large increases in hardness and strength can be obtained by cold-working, e.g., yield strengths of the order of 90 tons per sq. in. Further improvement in hardness and strength may be produced by a single short-time ageing at 800°-900°F. (425°-480°C.).

Corrosion-resistance of the new steel, in the condition obtained by sub-zero treatment and tempering, is claimed to be comparable, in some acid media, with that of molybdenum-containing nickel-chromium austenitic steel, and to be superior to that of straight 18-8 in glacial acetic and one per cent. sulphuric acids. Maximum resistance to attack by boiling 65 per cent. nitric acid (Huey test), i.e., to intergranular corrosion, is shown in the refrigerated condition before tempering. Carbide precipitation occurring in the steel after double ageing results in some deterioration in corrosion-resistance.

Cast Corrosion-Resisting Alloys of Iron-Nickel-Chromium Types

E. A. SCHOEFER: 'A Selection Guide to Corrosion-Resistant Cast Alloys.' *Machine Design*, 1954, vol. 26, Dec., pp. 178–86.

This article gives an account of the compositions and properties of the corrosion-resisting iron-nickel-chromium alloys standardized by the Alloy Casting Institute, U.S.A. Physical constants, mechanical properties, machining characteristics and other data are tabulated for the respective types, and the considerations involved in balancing the requirements for mechanical strength, cost, fabricating characteristics, and corrosion-resistance, in specific conditions of use, are discussed.

The alloys covered are shown in the table below.

Atmospheric Corrosion of Iron-Nickel-Chromium Alloys: Chart

A. DE S. BRASUNAS: 'Air Corrosion of Fe-Ni-Cr Alloys.' *Metal Progress*, 1954, vol. 66, Dec., p. 114 B.

Nomographic chart, based on test data reported by BRASUNAS, ET AL. in *Proceedings of the American Society for Testing Materials*, 1946, vol. 46, p. 870. The chart provides for approximate calculation of the corrosion-resistance of iron-nickel-chromium alloys containing carbon about 0·4, silicon 1·2, manganese 0·8 per cent. By use of the chart it is possible: (*a*) with known composition of the alloy and temperature of operation, to predict rate of corrosion, and (*b*) with known temperature and tolerable degree of corrosion, to select a suitable alloy composition.

Designation (Alloy Casting Institute) *	Corresponding A.I.S.I. Wrought Alloy Type †	Composition (%), balance Iron							
		C	Mn (max.)	Si (max.)	P (max.)	S (max.)	Cr	Ni	Other Elements
CA – 15	410	0·15 max.	1·00	1·50	0·04	0·04	11·5-14	1 max.	0·5 max. Mo ‡
CA – 40	420	0·20-0·40	1·00	1·50	0·04	0·04	11·5-14	1 max.	0·5 max. Mo ‡
CB – 30	431	0·30 max.	1·00	1·00	0·01	0·04	18-22	2 max.	—
CC – 50	446	0·50 max.	1·00	1·00	0·04	0·04	26-30	4 max.	—
CE – 30	—	0·30 max.	1·50	2·00	0·04	0·04	26-30	8-11	—
CF – 8	304	0·08 max.	1·50	2·00	0·04	0·04	18-21	8-11	—
CF – 20	302	0·20 max.	1·50	2·00	0·04	0·04	18-21	8-11	—
CF – 8M	316	0·08 max.	1·50	1·50	0·04	0·04	18-21	9-12	2·0–3·0 Mo
CF –12M	316	0·12 max.	1·50	1·50	0·04	0·04	18-21	9-12	2·0–3·0 Mo
CF – 8C	347	0·08 max.	1·50	2·00	0·04	0·04	18-21	9-12	Nb or Nb + Ta §
CF –16F	303	0·16 max.	1·50	2·00	0·17	0·04	18-21	9-12	1·5 max. Mo, 0·20–0·35 Se
CH – 20	309	0·20 max.	1·50	2·00	0·04	0·04	22-26	12-15	—
CK – 20	310	0·20 max.	1·50	2·00	0·04	0·04	23-27	19-22	—
CN – 7M	—	0·07 max.	1·50	**	0·04	0·04	18-22	21-31	Mo–Cu **

*Most standard grades listed are covered for general application by A.S.T.M. Specification A/296–49T. A.S.T.M. Specifications A/217–49T, A/351–52T, and A/362–52T also apply to some grades.

†Listed only for convenience in determining corresponding wrought and cast grades. Because cast alloy chemical composition ranges are *not the same* as wrought composition ranges, cast alloy designations should be used for proper identification.

‡Molybdenum not intentionally added.

§Nb 8 × C min., 1·0% max.; *or* Nb + Ta 10 × C min., 1·35% max.

**Several proprietary alloy compositions fall within the stated chromium and nickel ranges, and contain varying amounts of silicon, molybdenum and copper.

Determination of Molybdenum in Steels

See abstract on p. 32.

Reaction between Nickel and Molten Sodium Hydroxide

See abstract on p. 27.

Sulphide Corrosion Cracking in Oil-Well Equipment

NAT. ASSOCN. OF CORROSION ENGINEERS, TECHNICAL UNIT COMMITTEE 1-G: 'Sulphide Corrosion Cracking of Oil Production Equipment.' *N.A.C.E. Publn.* 54-5. *Corrosion*, 1954, vol. 10, Nov., pp. 413-9; disc., p. 419.

The report (prepared by R. TRESEDER) summarizes field experience accumulated by the N.A.C.E. Committee on Sulphide Corrosion Cracking of Production Equipment. It deals particularly with tubing and casing, in sour gas-condensate and high-pressure sour oil wells. Preventive measures are described. Materials exposed in some of the tests reported included A.P.I. N-80* steel, 5 and 9 per cent. nickel steels, 7 and 9 per cent. chromium steels, low - chromium - molybdenum and nickel - chromium-molybdenum types, high-chromium steel (carbon 0·15 max., chromium 11·5-13·5, per cent.); and austenitic steel of the A.I.S.I. 322 (precipitation-hardenable type).

See also—

J. P. FRASER and R. S. TRESEDER: 'Resistance of Tubular Materials to Sulphide Corrosion Cracking.' *Amer. Soc. Mechanical Engineers, Paper* 54-PET-20, Sept., 1954; 16 pp. + tables and figures.

A typical sulphide-corrosion-cracking failure of N-80* tubing in a sour gas-condensate well is described in detail, and an account is given of a laboratory test by which materials can be evaluated with regard to their susceptibility to this type of attack. Using this method, based on the critical stress required to cause cracking in a standard laboratory test, a study was made of the relative susceptibility of a large number of heats of commercial tubular materials, including N-80 and 5 and 9 per cent. nickel steels.

Materials for Handling Sulphuric Acid and Acid Sludges in Refineries

V. J. GROTH and R. J. HAFSTEN: 'Corrosion of Refinery Equipment by Sulphuric Acid and Sulphuric Acid Sludges.' *Corrosion*, 1954, vol. 10, Nov., pp. 368-89; disc., pp. 389-90.

The authors review the uses of sulphuric acid in the processing of oil stocks, and discuss the nature of the acid sludges formed at various stages.

The first part of the paper deals with general corrosion problems associated with tanks, lines, valves, pumps and heating coils; the second contains information on the resistance of various materials to corrosion in the conditions involved in such service, indicating the relative suitability of individual materials for specific types of equipment. The information given is derived from plant experience as well as laboratory tests. Recommendations are made also with regard to fabrication and design, maintenance, precautions required with welded and riveted installations, galvanic effects liable to be set up in service, and other variables which may affect behaviour and service life. The information on materials is conveniently summarized in a series of tables (1) reporting experience in refinery plant, and (2) giving corrosion-test data on individual metallic materials in contact with typical sulphuric-acid and sludge corrosives. The significance of temperature, velocity and turbulence, the presence of inhibitors or accelerators, galvanic action, and the concentration of the corroding medium is emphasized.

In general comment on materials which are suitable for use in the types of service under discussion, the authors briefly discuss some which are outstandingly useful: these include:—

Iron-silicon alloys, which have excellent resistance to erosion-corrosion attack at all concentrations of sulphuric acid up to 100 per cent. and at all temperatures up to and including boiling. Such alloys are, however, susceptible to damage from physical and thermal shock.

Lead, which is extensively used in handling sulphuric acid, at room temperature. Its corrosion-resistance depends on formation of a protective film. The limitations of concentration and temperature regulating the serviceability of lead are indicated.

Stainless steels of the 18-8 and 18-8-Mo types, used to only a small extent in sulphuric-acid service: their behaviour is strictly conditioned, within narrow limits, by the concentration of the acid solution. The more highly-alloyed types of chromium-nickel steel, containing more than 20 per cent. of each element, together with small amounts of copper, molybdenum, and silicon are, however, widely used, e.g., for valves. Such materials show satisfactory resistance to corrosion by all concentrations of sulphuric acid at temperatures below about 150°F. (70°C.) and are also usable with oleum or fuming acid.

Monel is widely used in sulphuric-acid service, due to its desirable combination of mechanical strength and corrosion-resistance. Typical curves show the behaviour of Monel in acid of various concentrations.

Nickel-molybdenum alloys (of the Hastelloy or Chlorimet types) are used for handling both the acids and the sludges in conditions too severe to be withstood by other materials. This class of alloy is one of the few which show generally satisfactory resistance at temperatures above 200°F. (95°C.).

High-alloy cast iron of the Ni-Resist type is suitable for handling sulphuric acid at low concentrations and temperatures.

Nickel-containing Materials in Shafts

See abstract on p. 31.

* Typical analysis: carbon 0·49, manganese 1·45, molybdenum 0·42, per cent.

Corrosion-Resistance of Cupro-Nickel Alloys

F. L. LAQUE: 'Corrosion-Resistance of Cupro-Nickel Alloys containing 10 to 30 per cent. Nickel.' *Corrosion*, 1954, vol. 10, Nov., pp. 391–9; disc., p. 399.

Cupro-nickel alloys of varying nickel content have, during recent years, come into extensive use, principally in the form of tubes for heat exchangers, where their outstandingly good resistance to attack by salt water used for cooling is a major advantage. More recently they have been widely adopted for tubing in boiler feedwater heaters, where they are superior to other copper alloys in maintaining strength at the elevated temperatures used in modern high-pressure steam power plant. A considerable mass of data has been accumulated on the behaviour of the alloys in both these types of application; the present author gives reference to some of the principal literature of the subject.

Success in marine and power-plant service has led to the introduction of the cupro-nickel alloys into the chemical-engineering industries, and the necessity thus arises for reliable data on the resistance of the alloys to attack by corrosive media other than cooling waters. The information recorded in this paper is intended to serve as a guide in selection of the cupro-nickel alloys for specific types of service: the data presented are based on systematic laboratory studies in selected media, and on plant tests of commercial alloys exposed under normal operating conditions. Particular emphasis is placed on the latter source of information.

As an introduction, the author discusses the basic character of alloys of copper and nickel, and the theoretical aspects relevant to the corrosion-resistance which may be expected from such materials. The effect of nickel content on corrosion by some common media is illustrated, and the superior resistance of the copper-nickel alloys to stress-corrosion cracking is demonstrated by comparison with the behaviour of some other copper-base alloys.

A detailed report is then made of experience gained, over a period of some twenty years, with specimens of cupro-nickel alloys of varying nickel content, exposed in many kinds of plant in the chemical and process industries. The specimens used were of commercial production, and contained the usual amounts of other elements, of which the most important, from its effect on corrosion-resistance, is iron. (The beneficial influence of small amounts of iron, on resistance to attack by salt water, has been the subject of several earlier papers, by the author and by other investigators: some notes on current views on optimum percentages for this element are given in the present paper.)

The major part of the report comprises tabular data, suitably annotated, on the behaviour of 90–10, 70–30 (and in some cases 80–20) grades of cupro-nickel. Information is given on resistance to waters other than salt waters, ammoniacal solutions, brines, alkaline solutions, petroleum products containing hydrogen sulphide, miscellaneous petroleum products, organic compounds, chlorinated solvents, sulphuric acid, halogens and halogen compounds, and paper-stock suspensions. In all cases details are given of the conditions of exposure, and the reference data thus provided constitute a valuable source of reference. They clearly indicate the value of the cupro-nickel alloys in many branches of the chemical industry.

Testing of Valves for Use in Corrosive Conditions

J. BOLTON: 'Accelerated Tests Aid Valve Selection.' *Chemical Engineering*, 1954, vol. 61, Nov., pp. 268, 270.

Attention is directed to a new test procedure which has been developed by the Lunkenheimer Company to assist the users of valves required to operate in corrosive conditions. The test simulates service conditions, in which the corrosive fluid is in constant motion over the valves.

Reference is also made to the successful use of austenitic nickel-containing cast iron as the valve body material, with Monel or nickel-chromium stainless steel as trim. Some typical data for service of such valves in sulphuric-acid solutions are given.

Production of Welded Nickel-Chromium Steel Piping

A. GRODNER: 'The Production of Welded Stainless Steel Pipe.' *Welding Jnl.*, 1954, vol. 33, Dec., pp. 1171–4.

In connection with the erection of the A.E.C. Atomic Hydrogen plant at Savannah River a large quantity of stainless-steel pipe was required for conveying gases, liquids, and radioactive waste products at high temperatures and pressures, and under corrosive conditions. The size of the piping called for was, in some cases, beyond the maximum size limits of seamless-pipe mills, and the need therefore arose for production of welded piping. This article describes methods jointly developed by the engineers of the Alloy Fabricators Division of Continental Copper and Steel Industries, Inc., of Perth Amboy, N.J., and of the Atomic Energy Commission.

The pipe to be produced ranged in size from 3 to 14 in., with wall thicknesses of $\frac{1}{8}$ in. for the smaller pipe up to $\frac{7}{16}$ in. for the larger size. In most cases the material used was extra-low-carbon 18–8 chromium-nickel steel (to A.I.S.I. 304L specification): for the remainder niobium-stabilized 25–12 chromium-nickel steel to Type 309 specification was employed. All the pipe was made in 10-ft. lengths.

This article describes, step-by-step, the production methods used, from receipt of the 'skelp' (annealed and pickled), through *forming* and *welding* ('sigma' consumable-electrode process); *radiographing* of the welds; postweld solution *heat-treatment* (1950°–2050°F.; 1065°–1120°C., for 15–30 minutes): checking of the pipe, for susceptibility to intergranular corrosion, by the *boiling nitric-acid test; pickling* (nitric-hydrofluoric acid solution); *hydrostatic pressure testing*, and *inspection*. After inspection, the pipe was finally passivated in a 30 per cent. nitric acid solution, at 130°F., (55°C.) and rinsed in warm water.

Testing of Stainless-Steel Welds

H. THIELSCH: 'Testing of Stainless-Steel Weldments.' *Welding Research Council Bull. No.* 18, Sept., 1954; 26 pp.

This is one of a series of bulletins in which the current stage of development and knowledge on various aspects of welding is being summarized, on the basis of the literature and of other available information. The *bibliography* supporting this review contains 150 items. The classification of the subject matter, which is essentially of a reference character, is shown in the skeleton of contents below:—

Welded Joints
- A. Special Welds (usually tested by destructive tests)
  - Tensile, impact and bend tests.
  - Fillet-weld tests.
  - Procedure-qualification tests.
- B. Production Welds (for which non-destructive tests are frequently used)
  - Fluorescent-penetrant, dye-penetrant, radiographic, or ultrasonic inspection.
  - Sectioning of welded joints.
  - Leak testing, by various methods.

Resistance Welds (spot, seam, projection, flash and percussion welds)
- Mechanical tests.
- Radiography.
- Metallographic tests.
- Magnetic inspection.

Corrosion Testing
- Definitions of intergranular corrosion, and knife-line attack.
- Scope and validity of corrosion tests.
- Individual corrosion tests applied to welds.
  - Boiling nitric-acid (Huey) test.
  - Boiling acid copper-sulphate tests.
  - Nitric-hydrofluoric acid test.
- Preparation of welded specimens for corrosion test.
- Effects of composition on corrosion-resistance.

Metallographic Tests

Ferrite Determination
- Microscopic procedures, etchants used, etc.
- Micromagnetic inspection.
- Magnetic inspection.

Carbide Determination
- Microscopic procedures, etchants used, etc.

Sigma Determination
- Etchants used, their selectivity, etc.
- Heat tinting.
- X-ray diffraction procedures.

Microhardness Tests

Weld-Metal Cracking
- Fillet-welded tee joint.
- Reeve test.
- Navy-patch test.
- Circular-groove test.
- Slotted-groove test.

As an example of specifications which have been developed by various code-writing and standardizing bodies for evaluation of stainless-steel electrodes, details are given of the conditions laid down in a specification drawn up by Royal Dutch Shell Laboratory, Holland.

Fatigue Strength of Spot-Welded Stainless Steels: Effect of Prestressing

A. CHOQUET, V. N. KRIVOBOK and G. WELTER: 'Effects of Prestressing on Fatigue Strength of Spot-Welded Stainless Steels.' *Welding Jnl.*, 1954, vol. 33, Oct., pp. 509S-23S.

This paper, reporting research carried out to provide design data for engineers using spot-welded stainless steel in structural applications, gives results of tests on welds made in nickel-chromium steel of the composition shown below, used in the half-hard temper, for which the properties shown in the table are typical.

The fatigue strength of some metals and alloys is often taken as a ratio to the tensile or shear strength of the material, but tests on spot-welded joints showed that such a criterion was not valid in this case.

The paper gives a closely detailed account of experiments in which fatigue tests were made on 1, 2, 3 and other multiple-spot-welded sheet specimens. Conditions were adjusted, in the respective tests, to reveal the effects of such variables as spot pattern, interspot distances, etc.

In addition, valuable information was obtained on the marked improvement obtainable in fatigue-resistance by pre-straining of spot-welds, by 'hydrostatic' compression, simple compression, tension, or hand peening. The greatest improvement is obtained by hydrostatic compression: fatigue strength progressively rises as the load is increased up to 400,000 p.s.i. (178·5 tons per sq. in.). Simple compression, tensile straining, or peening also effects substantial increase in fatigue strength.

C	Si	Mn	S	P	Ni	Cr	U.T.S.		Yield Strength 0·2% offset		Fatigue Strength	
%	%	%	%	%	%	%	p.s.i.	t.s.i.	p.s.i.	t.s.i.	p.s.i.	t.s.i.
0·11	0·63	0·73	low	low	6·70	17·80	168,000	75	128,500	57·5	100,000	44·5

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*

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